NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE

No. 1207

REACTIONS WITH STEEL OF COMPOUNDS CONTAINING CHEMICAL GROUPS USED IN LUBRICANT ADDITIVES

By Allen S. Powell

Aircraft Engine Research Laboratory
Cleveland, Ohio

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SUMMARY

The chemical reactions between steel of a type used in aircraft-engine cylinder barrels and compounds containing reactive groups commonly found in lubricant additives were investigated. The products formed by reaction at temperatures from 400° to 650° F were analyzed by reflection electron diffraction. The principal corrosive action found was caused by oxygen carried in the reactant as dissolved air or dissolved moisture. Additive reactivities were in agreement with predictions based on knowledge of the chemical reactivity of the groups and linkages tested. The reaction product could be identified when complication of the diffraction patterns by oxides of iron was eliminated. Compound formation observed in this investigation supports the theory of boundary-lubricant additive action, which states that additives form compounds of low shear strength at metal surfaces and prevent seizure and high abrasive wear when no fluid film is present.

INTRODUCTION

A review of the work already done in the field of lubrication of bearings under high loads indicates that a study of the chemical reactions between steel and lubricant additives would be of value in future considerations of the use of additive-containing oils in high-output aircraft engines. Wells and Southcombe (reference 1) first suggested the possibility of decreasing the friction of bearings operating under conditions of low speed and high load where a thick fluid lubricant film cannot be maintained. For these conditions of lubrication - generally termed "boundary conditions" - the addition of fatty acids to mineral oils was found to be an effective method of reducing coefficients of friction (reference 1). Investigations by Sir William Hardy (reference 2), Langmuir (reference 3), Beeck (reference 4), and Dacus, Coleman, and Roess (reference 5) have demonstrated that such long-chain polar-compound additives aid lubrication by promoting the maintenance of adsorbed

films of additive at the bearing surface. The interaction between additive and surface is entirely physical until temperatures and pressures at bearing surfaces become extreme (references 4, 6, 7, 8, and 9).

For the prevention of wear at bearing surfaces under extreme boundary conditions, Beeck, Givens, and Williams (reference 10) have found that long-chain polar compounds added to oils were relatively ineffective. Their work has led to the use of addition agents similar to tricresyl phosphate, which impart a high polish to a metal surface. The chemical experiments described in reference 10 showed that long-chain polar compounds together with these chemical polishing agents reduced wear by a factor of 2. The investigators proposed the theory that chemical polishing agents, such as they have worked with, react at the high temperature of a bearing under boundary conditions to form, by corrosive action and subsequent reduction by carbon, a metallic compound capable of alloying with the bearing surface; these compounds lower the surface melting point toward the eutectic temperature and thereby enable the surface to become polished. When the bearing surface is polished, its running temperature is lowered and the corrosive action of the polishing agent becomes negligible. Beeck (reference 4) states that the action of extreme-pressure additives in oils differs from the action of chemical polishing agents in that the extreme-pressure additives produce abrasive nonmetallic surface layers that prevent welding of the bearing metals when the lubricant film breaks down. A more general theory of boundary-lubricant additive action states that, when no fluid lubricant film can be maintained between the rubbing surfaces, additives prevent seizure and high wear of bearing surfaces by reacting chemically with the bearing metal to form an easily sheared nonmetallic layer on the surface (reference 11).

Davey (reference 12) has investigated the reduction of friction at extreme pressure by use of oils containing reactive sulfur or chlorinated compounds and has concluded that a chemical reaction is the source of their properties. This conclusion is well supported in the case of chlorine compounds by tests of load-carrying capacity (reference 13). The ability of a lubricant to carry higher loads was found to be associated with the chemical reactivity of the chlorine in the compound used as the additive. The reactivity was changed by changing the nature of the groups attached to the carbon bearing the chlorine. The load-carrying capacities of the lubricants could be predicted from theoretical knowledge of the chemical behavior of the chlorine compound thus formed. In a study of cutting fluids and the chemical and physical basis for their action, Shaw (reference 14) has presented evidence of chemical reaction between cutting

fluids similar to boundary-lubricant additives and aluminum. The surface compounds, which would be produced by chemical reactions between metals and the additive-type reagents that these workers tested, are nonmetallic and of lower shear strength than the metal itself.

The effect of the surrounding atmosphere on lubrication phenomena seems to be of great importance, although no investigation has been conducted to determine the basic reactions between lubricant, bearing surfaces, and atmosphere. Simard, Russell, and Nelson (reference 15) have investigated the reactions between bearing surfaces and oils or additives containing lead and sulfur. Oils containing active sulfur reacted with steel giving γ-FeOOH (rust), hydrated iron oxide, and Fe_3O_4. Oils compounded with lead naphthenate alone gave $PbSO_4$ and unidentified patterns (one of which might be a form of FeS). Lead naphthenate and sulfur together reacted to give $PbSO_4$ and PbS; the $PbSO_4$ formed first, apparently because of oxygen dissolved in the oil. The base oils gave γ-Fe_2O_3 and an unidentified pattern. (This pattern also might be a form of FeS). Simard, Russell, and Nelson (reference 15) conclude that the effect of the oils appears to be limited to their action as oxygen carriers or to possible interaction with the addition agents. Kinetic-friction tests by Gilson (reference 16) showed that the coefficient of friction of a lubricated bearing decreased in going from an evacuated space about the bearing to an atmosphere of air and decreased further when the atmosphere was changed to oxygen. The coefficient of friction in hydrogen was higher than in air. Addition of moisture to the hydrogen decreased the coefficient of friction. Bowden, Leben, and Tabor (reference 17) found that the motion of a steel slider on a steel plate changed from "stick-slip" to smooth sliding after the system was heated in an oxidizing atmosphere. The oil was probably changed by oxidation but no study of corrosive action was made. In the case of lubrication by graphite, the development of generator brushes to operate at high altitudes resulted in the discovery that the entire lubricating action of graphite arises from moisture adsorbed on the graphite particles (reference 18). Without an adsorbed film of water, graphitic carbon acts as an abrasive. These results indicate certainly that air and moisture are important aids to lubrication, but practical experience with highly loaded bearing surfaces definitely has shown a need for additives to supplement the oxygen normally carried in an oil.

The previous investigations discussed have shown that boundary lubrication under extreme conditions involves chemical reaction among bearing surfaces, surrounding atmosphere, and lubricant. For a more complete understanding of the reactions between bearings and lubricants

identification of the products formed and reaction-rate studies are necessary. Specimens obtained from a bearing test machine reproducing actual operating conditions are generally unsuitable for such studies because of lack of control of many variables during the tests. Nonuniform conditions of temperature, pressure, and surface structure exist during operation of a bearing. Several reactions are competing to form surface films. The nature of the lubricant and the surrounding atmosphere is variable. A machine allowing operation of bearing surfaces under controlled conditions of temperature, pressure, lubricant, surface material and finish, and surrounding atmosphere would give important information on friction and wear and would provide information of fundamental value. Chemical analyses of oil used in a machine of this kind for dissolved metals, oxidation products, or other changes are needed. Surface examination of the used bearings for physical changes and products of chemical reactions is also required.

The scope of the present investigation of additive action has been limited by eliminating as far as possible the variables of a bearing test in order to produce a single chemical reaction on a uniform surface at a known temperature. This elimination of variables was accomplished by carrying out the reaction between surface and test compound in a glass tube sealed under vacuum. Electron diffraction, which provides one of the best methods of studying surface chemical films, was used for the identification of the products formed during these tests. The reactants chosen for the experiments were steel of a type used in aircraft cylinder barrels and compounds containing reactive groups and linkages commonly found in extreme boundary-lubricant additives. The investigation was conducted at the NACA Cleveland laboratory during 1944 and 1945.

MATERIALS, APPARATUS, AND PROCEDURE

The additive-type compounds tested were:

Aliphatic linkage	Reactive group	Aromatic linkage
Stearic acid	Carboxyl	Benzoic acid
Tetrachlorethane	Chloride	Chlorobenzene
Dodecyl sulfide	Sulfide	Phenyl sulfide
n-Butylamine	Amino	Aniline

The test surfaces were of SAE 4140 steel metallographically polished with aluminum oxide on broadcloth. The compounds were tested at

temperatures from 400° to 650° F, which are in the range attained by aircraft-engine cylinders during operation, although probably lower than the temperature at the sliding surfaces. The specimens were examined before and after experiments by reflection electron diffraction.

A series of exploratory tests in this investigation revealed that a vacuum of less than 1 micron of mercury was required to remove oxygen from the reagents and evacuating system to such an extent that iron-oxide patterns were no longer prominent. The procedure used to attain this vacuum condition was evacuation of a tube containing steel and reagent by an oil diffusion pump sealed to the tube. When the pressure measured by a thermocouple gage was below 1 micron, or in the case of volatile compounds, when about one-half of the reagent had been pumped from the system, the glass tube containing reagent and specimen was sealed under vacuum. These sealed tubes were heated in bombs at the reaction temperature for 3 to 4 hours. After cooling, the tubes were opened and the steel specimen was washed in benzene and transferred with as little delay as possible to the diffraction camera.

The electron-diffraction camera used was the standard adaptor for the type EMB-4 RCA electron microscope. The patterns obtained were identified by comparison with the A.S.T.M. card file of X-ray diffraction patterns. The over-all precision of the method used for pattern measurement was 3 percent or better. With the use of estimated intensities, this precision permits almost positive identification of the surface films found.

RESULTS AND DISCUSSION

Because the compounds chosen for study contain the chemical groups typical of those used in lubricant additives in the extreme boundary-lubrication region, the following discussion is therefore limited to consideration of the lubrication process only under extreme boundary conditions. The results are summarized in table I and diffraction patterns typical of those from which the data of this table were obtained are shown in figure 1.

Figure 1(a) shows a diffraction pattern from a surface before test. The noteworthy features of this pattern are faint lines of the α-Fe pattern, broad lines at the positions usually associated with polished surfaces, and a great deal of background haze caused by specular reflection of electrons. These features indicate an undulatory roughness and some scratches and pits remaining on the

polished specimens; these conclusions were confirmed by microscopic examination of the polished surfaces.

The results of these experiments are in excellent agreement with the known facts about the groups and linkages tested. Benzoic acid is a stronger acid than stearic acid. After being heated for 3 to 4 hours at a temperature of 400° F in a sealed tube with stearic acid, a polished steel specimen had etched to a matte gray. The electron-diffraction pattern from the specimen proved to be a mixture of the patterns of α-Fe and α-Fe_2O_3. Benzoic acid at 400° F etched the surface more deeply and gave only the pattern of α-Fe. The corrosive action of benzoic acid was intensified at 500° F. The occurrence of α-Fe_2O_3 in the reaction with stearic acid could be due to dissolved air in the acid inasmuch as the reaction tube containing the specimen and acid was evacuated at room temperature. Iron stearate or benzoate, which might have formed on the steel surfaces, would have been removed along with the unreacted acids when the specimens were washed with benzene.

The chlorine-containing compounds, tetrachlorethane and chlorobenzene, were used at temperatures of 400°, 500°, and 650° F. The compounds found on the steel specimens were FeOCl and $FeCl_3$ in the case of tetrachlorethane, and Fe_3O_4, in the case of chlorobenzene. The Fe_3O_4 was taken as an indication of residual moisture in the chlorobenzene used. FeOCl results from hydrolysis of $FeCl_3$ during transfer of the specimen from the reaction tube to the diffraction camera. When the specimens were briefly exposed to air, $FeCl_3$ lines disappeared from the pattern obtained and β-FeOOH lines appeared as the hydrolysis progressed. The failure of chlorobenzene to form a chloride with iron is to be expected as typical of the low reactivity of halogen in an aromatic linkage, whereas the formation of $FeCl_3$ by tetrachlorethane shows the high reactivity of the aliphatic chloride linkages.

Dodecyl and phenyl sulfides were used for the tests of the sulfide group. After heating at 500° F, dodecyl sulfide gave a specimen coated with a black material that gave the diffraction pattern of FeS. Phenyl sulfide, on the other hand, reacted at 550° F to form principally FeS_2, although lines tentatively identified as arising from a form of FeS also appeared in the diffraction pattern. Theory would predict a lesser reactivity for phenyl sulfide than dodecyl sulfide because of the greater bond strength between carbon and sulfur in an aromatic linkage than in an aliphatic bond. Formation of FeS_2 would require a more reactive sulfur than

formation of FeS. A possible explanation is that the phenyl sulfide used contained polysulfides as impurities. The only other means by which phenyl sulfide could react with steel would be through formation of biphenyl.

The amino-group compounds tested were n-butylamine and aniline. At temperatures of 400° , 500° , and 650° F, n-butylamine gave an unidentifiable compound and Fe_3O_4 on the specimen surface. Aniline was run only at 450° F, at which temperature a mixture of γ-Fe_2O_3 and α-Fe_2O_3 formed on the surface. The presence of oxides on steel after heating in amines is somewhat unexpected as amino compounds are added to oils as antioxidants. No reaction between amines and steel was expected, but the reducing action of the amines would have been expected to prevent the oxidation of the steel specimens. Theoretically, n-butylamine would be a better reducing agent than aniline because of the stabilization of the amino group of aniline by its bond to an aromatic nucleus.

The formation of γ-Fe_2O_3 is characteristic of the low-pressure and low-temperature oxidation of steel by air (reference 19); α-Fe_2O_3 is formed from γ-Fe_2O_3 by transformation at approximately 650° F under normal conditions. The presence of γ-Fe_2O_3 after heating in aniline suggests that the aniline used contained dissolved air. The formation of Fe_3O_4 by heating steel in n-butylamine might be due to residual moisture in the amine inasmuch as prolonged drying of n-butylamine over potassium hydroxide decreased the intensity of the Fe_3O_4 pattern.

Because γ-Fe_2O_3 and Fe_3O_4 are isomorphous, the diffraction patterns were differentiated on the basis of the sharpness of the diffraction rings in accordance with the statement of reference 15 that diffuse diffraction patterns are typical of γ-Fe_2O_3. Comparison of diffraction patterns obtained from surfaces heated in aniline and n-butylamine (figs. 1(d) and 1(e)) exemplifies this difference in sharpness. Exposure to air during transfer from reaction tube to camera may contribute to the oxide patterns found.

The action of oxygen as an aid to lubrication has been investigated and discussed in references 16 and 17. The fact that the presence of oxygen led to the formation of iron oxides in preference to the reaction between the additive-type compound and steel, as shown by the obscuring of the reaction-product diffraction pattern, indicates that under ordinary conditions oxygen carried in a lubricant can be an important aid to lubrication. The diffraction patterns obtained

in reference 15 supported the conclusion that in extreme boundary lubrication the effect of oils was limited to their action as oxygen carriers or to possible interaction with the addition agents. This conclusion neglects the consideration that neither the experiments of reference 15 nor the experiments reported herein extended to reaction conditions where the rate of reaction between additive and steel is much greater than the rate of oxidation of steel. The additives probably become important when oxygen is no longer available at the lubricated surface, as is shown by the present results, although bearing-test samples suitable for diffraction examination cannot be obtained from tests under conditions where the action of oxygen has been eliminated. Removal of oxygen by reducing agents, such as amines added to oils, may be somewhat harmful inasmuch as oxygen actually serves as an aid to lubrication. Ordinarily this effect is neutralized by using a second reactive additive to counterbalance the loss of oxygen. The most important property of an additive is undoubtedly the nature and ease of formation of the product of reaction between surface and additive. Unfortunately no direct correlation of the results of this investigation with friction and wear measurements is available.

If the reactivity between additive and steel is assumed to be the reason for friction and wear reduction by oil additives, heat-resistant and corrosion-resistant steels, such as the SAE 4140 steel used in these experiments, require more active lubricant additives than cast-iron or low-alloy steels to insure the same degree of lubrication. Further investigation is needed to establish this theory, but the assumption on the basis of the results of this investigation and the work discussed earlier in this paper seems reasonable.

Inasmuch as any compounds of iron except certain intermetallic compounds are of lower shear strength than metallic iron, the observed formation of nonmetallic surface films by reaction of additive-type compounds with steel is in agreement with the theory that additives aid lubrication by the formation of easily sheared contaminating layers on bare metal surfaces. Such layers enable bearing surfaces to slide over one another without seizure in the momentary absence of an oil film.

Correlation of the results of this investigation with the process occurring in a bearing is limited because only a few of the operating variables were reproduced. The additive-type compounds used were of technical purity, because compounds of this purity were available and actual additives used in compounding lubricants would also be of technical grade. Impurities such as dissolved air and moisture, which may be present in technical-grade reagents, tend

to mask the reactivity of the type compound in which they occur. In a dynamic system such as a journal rotating in a bearing at high temperature and load, reaction products from the impurities would be removed from the surface until the amount of reaction product formed became negligible, unless the impurity were renewed from an external source. The reactivity of metal surfaces when freshly formed, as in the case of abrasion due to running, is known to be greater than that of surfaces exposed to air and materials commonly present in air, such as water and oil vapors. Surfaces prepared, as in this investigation, by mechanical polishing are known to be of less activity than rougher machined surfaces.

The experiments of this investigation have been limited to chemical reactions between a few types of organic compound and steel. No precise correlation with reduction in friction, rate of wear, and load-carrying capacity has been attempted at this preliminary stage. By analogy, however, some inferences as to the relation between chemical reaction and other lubricant properties are justified and accordingly have been discussed.

CONCLUDING REMARKS

The chemical reactions between lubricant-additive-type compounds and steel were investigated by electron-diffraction examination of specimens heated in sealed tubes with the corrosive reagents. The following conclusions may be drawn from these experiments:

1. Compounds of the type used in lubricant additives reacted with steel surfaces at temperatures from 400° to 650° F to give identifiable products.

2. Additive reactivities with steel were in general agreement with predictions based on knowledge of the chemical reactivity of the groups and linkages tested.

3. Under the conditions used in these experiments, oxygen and water dissolved in the reagents tended to corrode steel more readily than the reactive group. When present, the oxide patterns obscured the diffraction patterns formed by other corrosion products.

4. The observed compound formation supports the theory of boundary-lubricant additive action, which states that additives form compounds of low shear strength at bearing surfaces and prevent seizure and wear when no fluid film is present.

Aircraft Engine Research Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, September 20, 1946.

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TABLE I - PRODUCTS OF CORROSIVE ACTION OF LUBRICANT-ADDITIVE-TYPE COMPOUNDS ON SAE 4140 STEEL

Compound	Temperature (°F)	Products	Probable process of formation
Aliphatic Linkage			
Stearic acid	400	α-Fe α-Fe_2O_3	Etching Dissolved air
N-Butylamine	400, 500, 650	Fe_3O_4 Unidentified product	Dissolved water - - - - - - - - - -
Tetrachlorethane	400, 500, 650	$FeCl_3$ FeOCl	Direct reaction Hydrolysis of $FeCl_3$
Dodecyl sulfide	500	FeS	Direct reaction
Aromatic Linkage			
Benzoic acid	400, 500	α-Fe	Etching
Aniline	450	γ-Fe_2O_3, α-Fe_2O_3	Dissolved air
Chlorobenzene	400, 500, 650	Fe_3O_4	Dissolved water
Phenyl sulfide	550	FeS_2, FeS	Direct reaction with impurities

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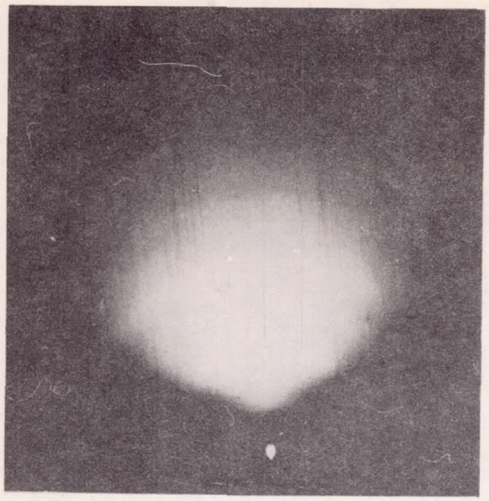
643



(a) Polished specimen before test. α-Fe; polish rings.

(b) Reactant, stearic acid; temperature, 400° F; compound formed, α-Fe_2O_3.

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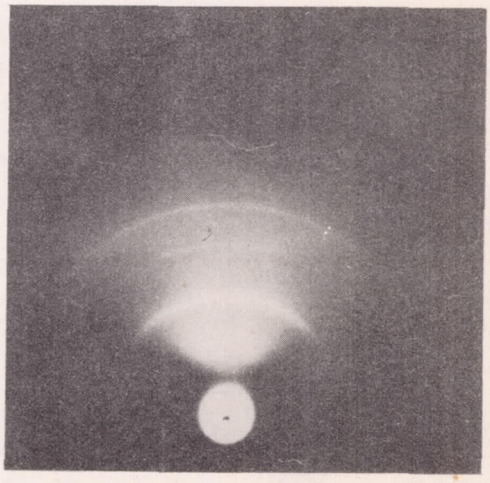
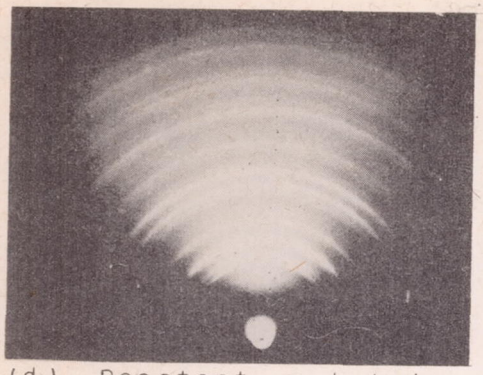


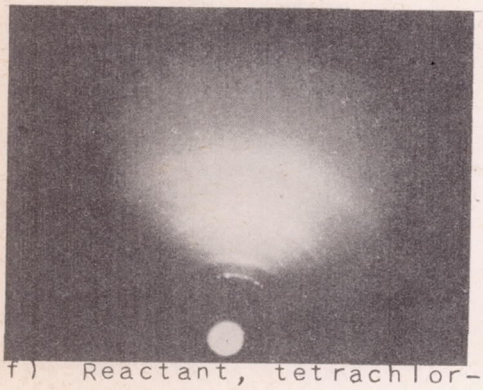
(c) Reactant, benzoic acid; temperature, 400° F; compound formed, α-Fe.

Figure 1. - Electron-diffraction patterns from SAE 4140 steel surfaces before and after heating with lubricant-additive-type compounds.



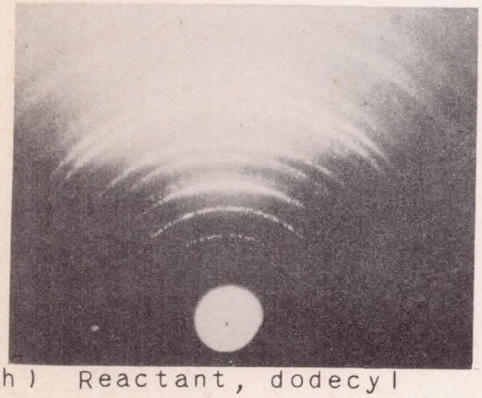
(d) Reactant, n-butylamine; temperature, 400° F; compound formed, Fe_3O_4.

(e) Reactant, aniline; temperature, 450° F; compound formed, α-Fe_2O_3; γ-Fe_2O_3.



(f) Reactant, tetrachlorethane; temperature, 650° F; compound formed, $FeCl_3$; FeOCl.

(g) Reactant, chlorobenzene; temperature, 650° F; compound formed, Fe_3O_4.



(h) Reactant, dodecyl sulfide; temperature, 500° F; compound formed, FeS.

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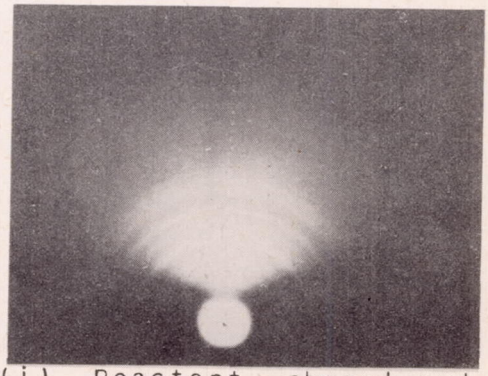

(i) Reactant, phenyl sulfide; temperature, 550° F; compound formed, FeS_2.

Figure 1. - Concluded. Electron-diffraction patterns from SAE 4140 steel surfaces before and after heating with lubricant-additive-type compounds.